REPORT DOCUMENTATION PAGE

AFRL-SR-AR-TR-03-

00839

1. REPORT DATE *(DD-MM-YYYY)*	2. REPORT TYPE	3. DATES COVERED (From - To)
July 2002	Final	5/01/2001 – 7/31/02

4. TITLE AND SUBTITLE

Acquisition of a High-Resolution Field Emission Electron Microscope For Nanoscale Materials Research and Development

5a. CONTRACT NUMBER

5b. GRANT NUMBER
F49620-01-1-0420

5c. PROGRAM ELEMENT NUMBER

6. AUTHOR(S)

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5d. PROJECT NUMBER

5e. TASK NUMBER

5f. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

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8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES)

10. SPONSOR/MONITOR'S ACRONYM(S)

11. SPONSOR/MONITOR'S REPORT NUMBER (S)

12. DISTRIBUTION / AVAILABILITY STATEMENT
Approved for public release; distribution unlimited.

13. SUPPLEMENTARY NOTES

14. ABSTRACT
The abstract can be found on page 2.

15. SUBJECT TERMS

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT	b. ABSTRACT	c. THIS PAGE			James M. Fitz-Gerald
unclassified	unclassified	unclassified	UU		19b. TELEPHONE NUMBER *(include area code)* 434 243 8830

ABSTRACT

Funds are requested for the purchase of a quantitative field emission gun (FEG) scanning electron microscope (SEM) at the University of Virginia (UVa). This quantitative microscope will contain a 25 kV Schottky FEG, energy dispersive x-ray spectrometer (EDS), in-situ nano-lithography system, chemical mapping and orientation imaging and strain analysis capabilities. The instrument will allow quantitative imaging and compositional analyses to be performed on all types of materials with high spatial resolution at low accelerating voltages (< 2.5 nm at 1 keV, for example), and it is essential for many current materials research programs within the School of Engineering and Applied Science (SEAS) at UVa. The increased resolution that a FEG SEM delivers will be a centerpiece for future research and development and characterization of nanoscale materials ranging from metals, electronic, and biological materials within SEAS and the College of Arts and Sciences, as well as for nearby academic institutions and industries. The new microscope is required to replace the existing SEMs, which are seriously outdated. It is clear that the new state-of-the-art SEM is required as a starting base in order to develop new materials in the nanoscale class that the DoD and the rest of the research community are pushing towards. The current SEMs have effectively served UVa in an efficient manner, providing over 15 years of continuous use for materials research. UVa has faculty and staff with sufficient expertise in SEM techniques to ensure that the new instrument is utilized for the highest quality materials research and that it is maintained at an optimum level of performance. The current track record that UVa has with other analytical instruments in the past are a strong testament to this infrastructure and we are confident this will continue in the future.

INTRODUCTION

The emphasis of materials science is to understand and control the processing-structure-properties relationships that exist in all classes of materials, with a current emphasis at the atomic-nanometer scale. In order to understand materials, it is necessary to quantitatively characterize the composition, structure, distribution and morphology of the component phases at many levels of detail. Since important compositional and structural variations can occur within very small regions (as in the small pore size of a nanotube, for example), complete characterization requires instruments capable of obtaining spectroscopic, morphological, and orientation information at the highest levels of resolution. For research in the areas of biological and polymeric materials, the damage to the sample from the electron beam is a clear concern. At present, an FEG SEM is one of the few tools capable of providing this information. It provides a critical link between observations that can be made below a few nanometers by transmission electron microscopy (TEM) and tens of nanometers by traditional SEMs and microns by light optical microscopy.

UVa has a history of excellence in materials research involving extensive collaboration among investigators within the School of Engineering and Applied Science (SEAS), including the Departments of Materials Science & Engineering (MSE), Electrical Engineering (EE), Chemical Engineering (CHE), Mechanical, Aerospace and Nuclear Engineering (MANE), as well as from Physics and Chemistry in the College of Arts and Sciences. Much of this research relies on the use of instruments in the Electron Microscope and Image Processing Facility located in MSE. The current facility has two JEOL Model 840 SEMs utilizing thermal emission filaments (W and LaB_6). The LaB_6 instrument has additional capabilities of wavelength dispersive spectroscopy (WDS), while the W filament instrument is a general use machine. The spatial resolution on the LaB_6 instrument, which is the better of the two, is 6 nm at 30keV on an ideal sample (for example, Au nanocrystals on C), but much less on fracture surfaces and typical materials research situations that are at the 2-5nm scale. In addition, neither of the current systems have modern software capabilities for orientation imaging, chemical mapping, image capture and analysis, etc, although the remainder of the microscope facility at UVa is highly digital. These combined deficiencies do not allow researchers at UVa to investigate material structures, compositions, and behavior at the level of a few nanometers, which is so critical to the research efforts put forth in the DoD and research community as a whole at this point in time.

The proposal was accepted and the equipment purchases were authorized over the period from 5/01/01 – 5/31/03 at a total cost of $720,000. The equipment installation is in the final stages of completion, with full sign off of the electron micrscope by the University of Virginia expected in the next 3-4 weeks. Three University of Virginia sources provided $108,000, and the DURINT provided $612,000. The next value of the new microscope and the upgraded instrument is $1,000,000 and its life is expected to be approximately 15 years.

Equipment Acquisition

The new state-of-the-art system is required to meet the challenges as a starting characterization base in order to develop new materials in the nanoscale regime that answer the many questions in terms of science, applied research, and the vision of the 20/20 campaign. In addition, it is tantamount that the required system be able to span all material classes, while maintaining the highest degree of resolution and analytic capabilities available today. An ultra high-resolution field emission gun scanning electron microscope (FEG SEM) is critical to current and future research, development and characterization of all material classes ranging from metals, electronic, and biological materials within SEAS and the entire University.

Critical Areas of Concern in Terms of Microscope Selection?

1) "The new FEG SEM and upgraded lithography system would have extensive applications to this program. When fabricating printheads with feature dimensions on the order of the tens of nanometers, an inspection tool with resolution of nanometers is critical. The FEG SEM is the most efficient and practical tool for providing such resolution. In particular, the ability to image at high resolution with low beam voltage (~ 1 kV, to reduce specimen charging and beam damage) is crucial for imaging the elastomeric molds that are a critical component of the microcontact printing techniques we employ. Such molds are difficult to image in the atomic force

.microscope, because the scanning tip is liable to deform the elastomer during imaging and distort the features, and specimen preparation for transmission electron microscope is impractical."
2) "The current SEM was incapable of effectively imaging the micro void processes at 20-100 nm diameter particles in either alloy class; the models remain speculative pending the application of state-of-the art analysis with an FEG SEM."
3) "It is *clear* that the addition of the new high-resolution FEG analytical SEM will have immediate positive impact in all four of the research areas outlined above, and may lead to further advances in that are of interest to the DoD with interaction of the many research scientists associated with this MURI, as outlined in the Research and Education in Section 6 to follow."
4) "The use of a high-resolution FEG SEM was the only viable method for accurate, high-resolution morphological characterization of polymer and electronic materials in the above research areas. Chemical sensors for biological warfare detection require nanometer thick films with controlled chemistry involving complex molecules such as SXFA (Poly(oxy{methyl[4-hydroxy-4,4,bis(trifluoromethyl)but-1-en-1-y1}silylene})) to be successfully integrated into useful DoD sensor systems. Using the FEG SEM allowed the investigation of properties such as surface roughness and morphology at high resolution without destroying the sensitive chemical polymer. In addition to chemical sensing, the use of the FEG SEM had significant impact project in the areas of conformal microelectronics and patterning of high-resolution phosphor display systems for heads up display systems (HUDS). In the areas of basic/applied research, the new microscope would enable the PI to continue research on carbon nanotube composites which require the use of an FEG SEM in terms of resolution and charging behavior."

From the above research description and concern areas, it is clear that an advanced microscope is needed with wide ranging capabilities to meet the above challenges. While these machines all have many options (literally hundreds), areas listed below are critical to our specific research mission, as outlined in the awarded proposal from the AFOSR. We feel that the JEOL model 6700F meets the criteria for the exhaustive research needs. These areas will be contrasted with other manufacturers later in this discussion.

1) Sample resolution at short working distances: 1nm @ 15 kV is the highest specified resolution currently available.
   This allows the scientist to resolve a feature on a conducting surface down to 1 nm in size at high voltages, while observing non-conducting samples down to 2.2 nm in size at low voltages. This is critical to the breadth of use in systems ranging from metals to insulating materials.
2) Faraday cup measurement in the column: This means that there is no need to observe the stage current reading. But it allows for input specimen current to be monitored, AND an optional specimen current detector can be added to the stage, thereby totally characterizing the specimen's current state at all times.
3) Aperture control lenses in column: This allows for better overall resolution (see 1 above), and simplified alignment.
4) Retractable solid-state backscatter: This feature allows for backscatter imaging at extremely short working distances (2-6 mm), which is critical to metal fracture surface analysis, reconstruction, and high -resolution mapping.
5) Large airlock: This allows samples up to 2" tall/thick to be examined which further enhances the ability of the machine to serve SEAS and the University, where the variety of sample is expected to vary with the incoming research focus of the 20/20 commission
6) Cold cathode field emission source: This means that the source of electrons for the purpose of imaging, analysis, and beam writing is from a cold field source. This source typically yields a small beam size with control at low accelerating voltages, which is a requirement for biological, insulating, and polymeric material specimens.

Manufacturer Compatibility

The Department of Materials Science and Engineering has been working closely with the JEOL corporation for over 20 years. Throughout the 20 years we have established the following professional relationship that has proved invaluable in terms of scientific research output and personnel:

1) Equipment: The department currently houses five electron microscopes manufactured by the JEOL corp.
2) Service: The JEOL corp. has provided outstanding service on all instruments
3) Service personnel: The service personnel representing JEOL corp. is a top qualified professional in his field who interacts well with the students, faculty, and technical staff in the MSE Dept.
4) MSE Technician Supervisor, Dr. Shian Chen: Dr. Shian Chen has 12 years of experience working with JEOL microscopes and we have a large investment in terms of time and money in his training and student education on JEOL instruments.
5) A provision in the award allows for a 200K$$ component upgrade of an older electron microscope manufactured by JEOL corp., model JEOL 840 scanning electron microscope. This upgrade will be better affected by the current JEOL technician, in concert with our MSE Supervisor (Dr. Shian Chen), due to the invested time in servicing and operating the electron microscope to be upgraded. Upgraded components consist of the following:

i. Analysis capabilities (EBSD)
ii. In-situ nano-lithography system (NPGS)
iii. State of the art energy dispersive x-ray detection system (EDS) with digital imaging
iv. X-Y stage retrofit and software control

Systems Purchased

**Items 1-10 are applicable to the new electron microscope, while items 5-7 are upgrades / retrofits for the current / old model Jeol 840 electron microscope. Item #8 is applicable to both systems for proper sample preparation.

Item	***Price***
1) New JEOL Model 6700F Scanning Electron Microscope	$440,000.00
2) Tilt and axis movement (5 axis automation)	$13,500.00
3) Robinson backscatter electron detector for elemental imaging and analysis	$15,850.00
4) SEIKO turbo pump (dry system)	$23,900.00
5) Edwards scroll pump	$11,625.00
6) Digital scan converter	$5,650.00
7) Chamberscope	$5,100.00
8) Gatan cold stage SEM module	$30,000.00
9) PGT Spirit EDS system	$65,000.00
10) Gatan MonoCL3 cathodoluminescence imaging and spectroscopy system	$105,500.00
Upgrades to Existing JEOL 840 System:	
11) Nanometer Pattern Generation System (NPGS-60)	$56,000.00
12) PGT Spirit EDS system	$65,000.00
13) Spirit stage automation system	$16,250.00
14) DEBEN beam blanking system	$22,000.00
15) HKL EBSP upgrade to channel 5 software	$25,330.00
16) Multi-scan interface switch	$1,300.00
Applicable to both microscopes:	
17) Gatan, PECS 681 high vacuum coater, applicable to both machines	$54,000.00
Total:	$956,000.00
Total, less discount from JEOL:	**$720,000.00**

Equipment Installation

The following four figures illustrate the installation, near final configuration and example imaging of new and upgraded microscopes.

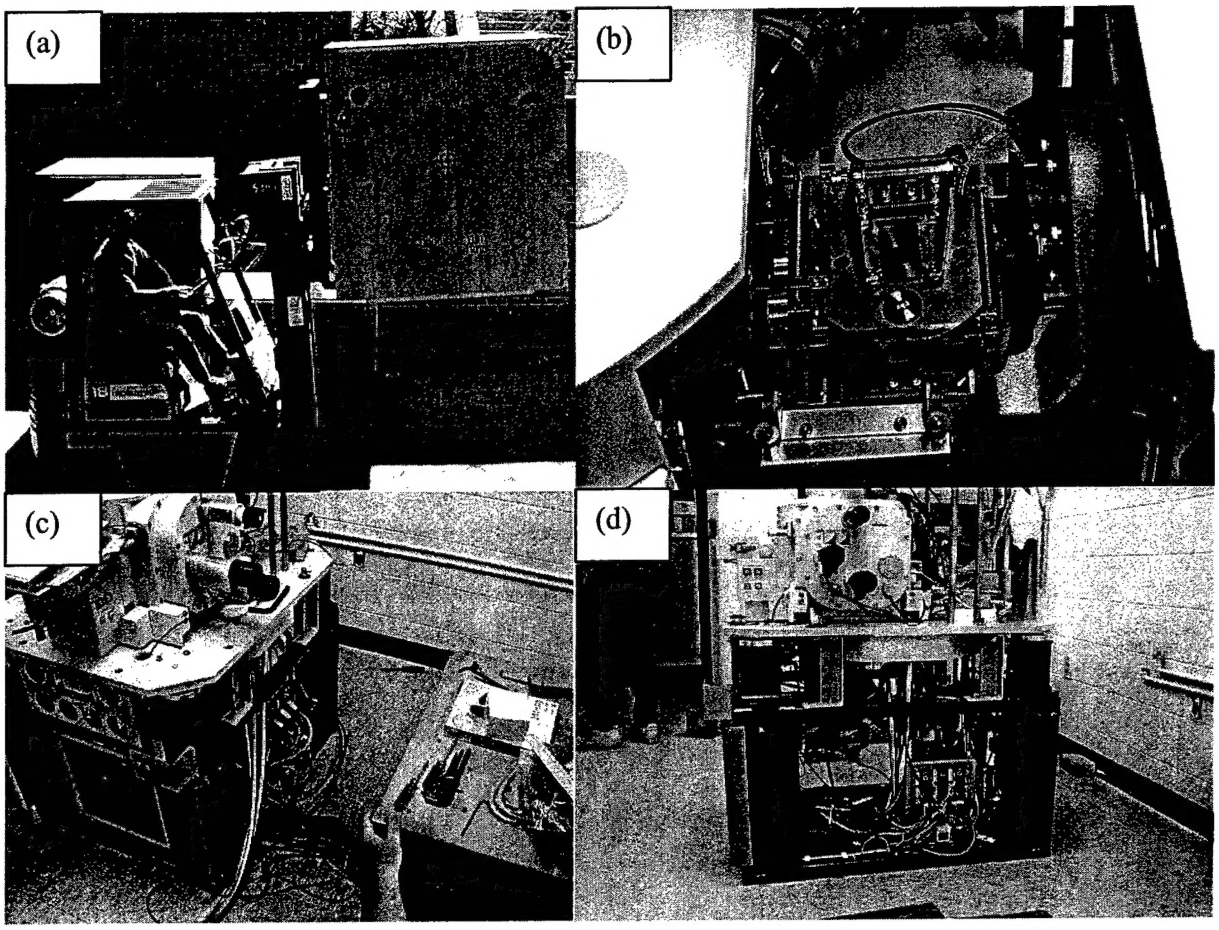

Figure 1. Delivery and various stages of installation of the JEOL 6700F scanning electron microscope.

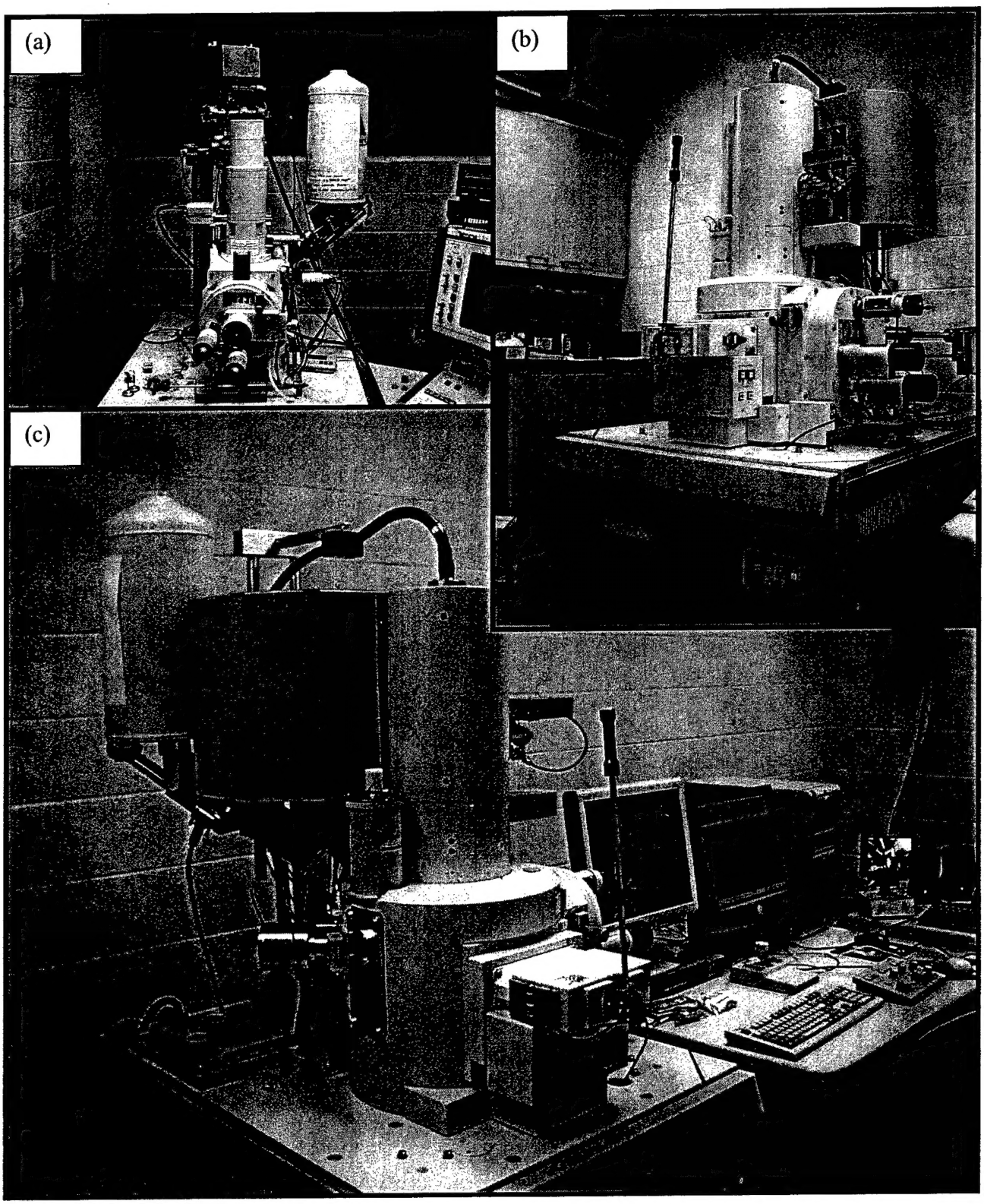

Figure 2. Near-final installation configurations for both the upgraded JEOL 840 machine (a), and the JEOL 6700F (b), (c).

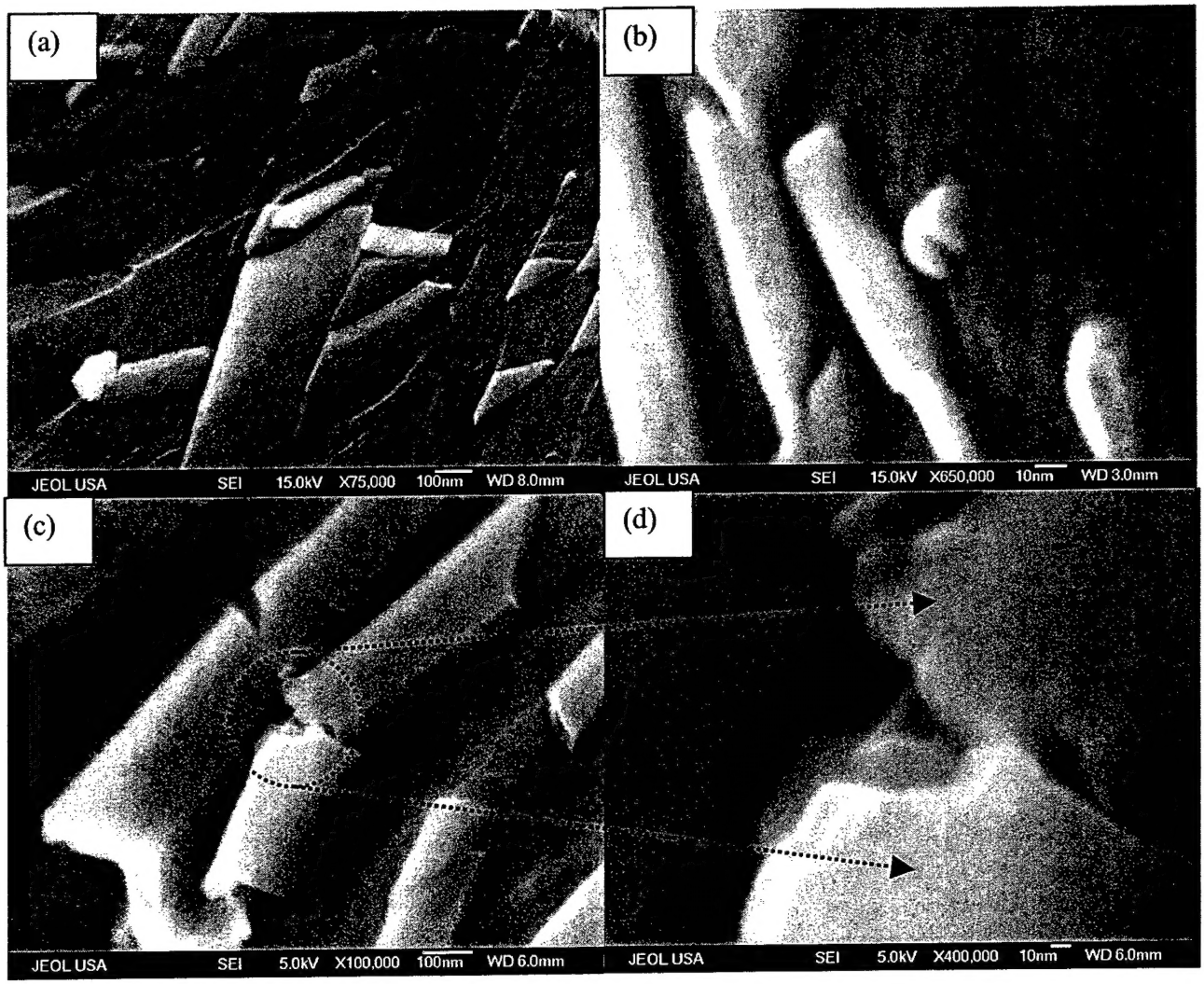

Figure 3. High-resolution SEM images using the in-lens detector (a-d) of a Al-Zn-Si-Cu fracture surface taken in the JEOL 6700F.

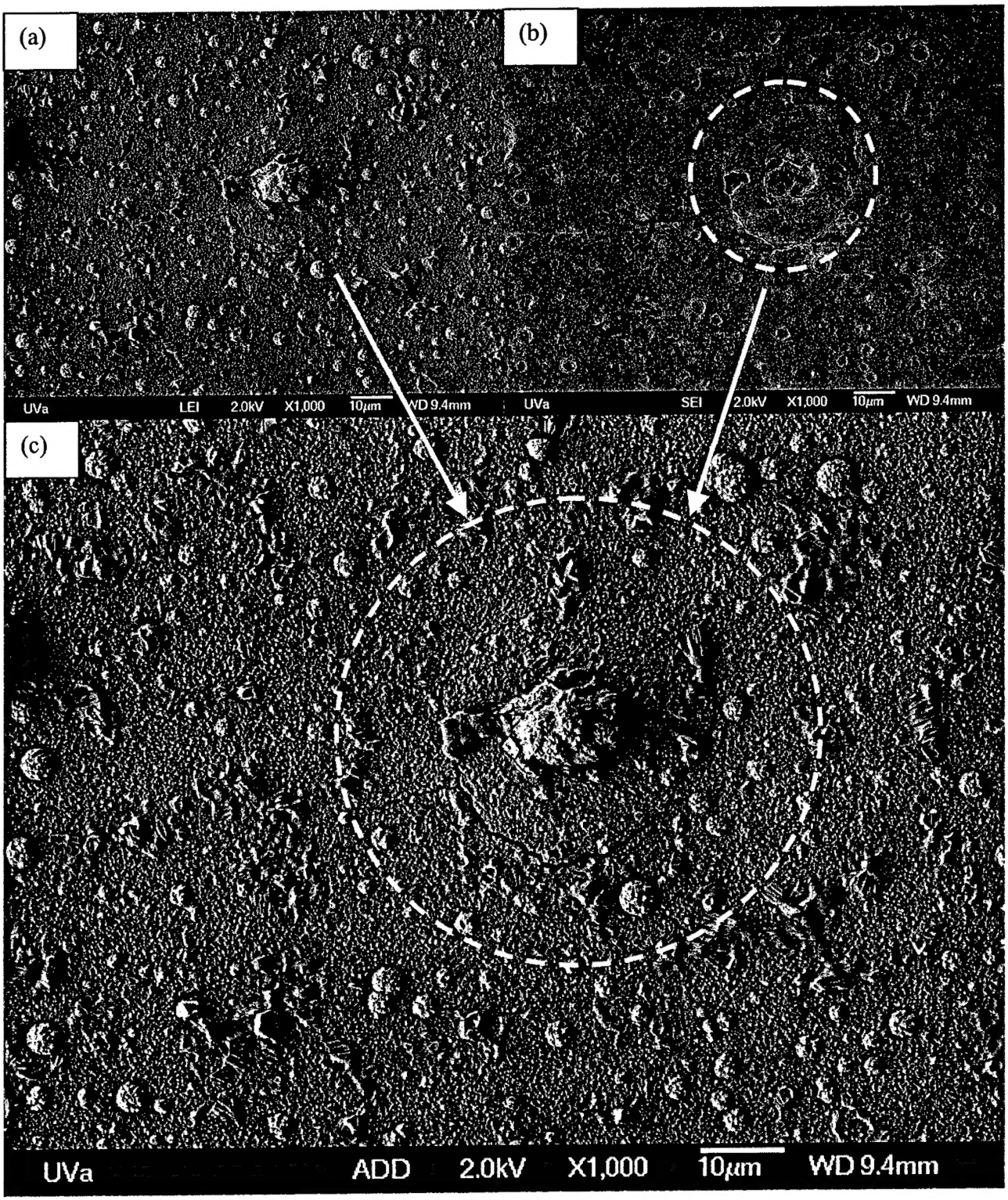

Figure 4. Scanning electron micrographs of SrS thin films illustrating the use of the two types of secondary electron detectors on the 6700F. Imaging achieved using the lower (conventional) and upper (in-lens) detectors separately is shown in (a) and (b) respectively, along with the combined image in (c). At this working distance, the in-lens detector gives very little morphological information, only contrast, while the converse is true for the lower detector. At working distances below 7-8 mm, the lower detector is not efficient, thereby realizing the importance of the in lens system for high resolution as in Figure 3.

Research Usage of Equipment

The intent of this section is to provide an illustration of the current and prior research that has been done at UVa with DoD support, and to show what is expected for the future of these areas in nanotechnology, with emphasis on DoD supported research. These examples are non-encompassing, and they should be treated as a "tip of the iceberg" image.

For the past decade, Richard P. Gangloff and his graduate students have conducted research on the mechanisms of fatigue and fracture of advanced structural alloys, as supported by grants from ONR ("Fundamental Studies of Occluded Crack Geometry Effects on Environmentally Assisted Cracking in High Strength Titanium Alloys", $161,961, 6/1/97 to 8/30/00, N00014-97-1-0523, with J.R. Scully; "Laboratory for Advanced Characterization of Environmental Fatigue and Fracture", $245,210, 3/31/99 to 3/31/01, N00014-99-1-0644; "Mechanisms of Crack Tip Hydrogen Embrittlement in High Strength Alloy Steels for Marine Applications", $389,998, 7/1/98 to 6/30/01, N00014-98-1-0740, with J.R. Scully), NASA ("Damage Tolerance of Light Aerospace Alloys Under Demanding Mechanical and Electrochemical Conditions", $389,300, 1/1/99 to 12/31/00, NAG-1-2176, with R.G. Kelly and J.R. Scully; "NASA-UVa Light Alloy and Structures Technology Program, 1/1/87 to 12/31/98, $7,500,000, NAG-1-745, with E.A. Starke and 6 other faculty), and industry (Alcoa, Boeing, General Electric, Newport News Ship Building and Dry Dock Company). This research has aimed to understand complex time-dependent damage processes localized to the tips of cracks in next-generation structural alloys and composites. Such understanding evolves from high-resolution characterizations of cracking, coupled with modeling from the micromechanical and microchemical perspectives. Results provide a necessary foundation for optimization of the reliability and durability of high performance alloys that operate in challenging mechanical and environmental environments. The new FEG SEM is critically important to propelling this research through current barriers. The new equipment will enable ultra-high resolution and quantitative analyses of damage processes in advanced structural alloys typified by complex microstructures at the 10-to 500 nm scale. This equipment will provide the means to probe the mechanical behavior of nanostructures defined according to DURINT Topic #8.

The results of the cited DoD and NASA-funded research illustrate the impact of the new FEG SEM. In the area of fatigue, crystallographic damage mechanisms abound, but are poorly understood particularly in fine-scale and complex microstructures. Electron Backscattered Pattern (EBSP) analysis provides a means to test experimentally various slip and cleavage-based hypotheses of damage evolution, as illustrated by the definitive demonstration of fatigue cracking parallel to {111} slip planes in Al-Li-based alloys fatigued in vacuum. In contrast, hydrogen-environment enhanced fatigue progressed along facets that were parallel to high-index planes and inconsistent with classical theories of deformation-nucleated brittle cracking. Results to date have been limited to the 5000 nm scale, and the hydrogen cracking mechanism is uncertain. A modern FEG SEM, with a state-of-the-art EBSP system, will yield crystallographic orientation information at the 100–1000 nm level and provide automation capabilities to improve the statistics of the measurements. Considering fracture, a challenge is to extend fractographic techniques to the 20–100 nm level. Micromechanical modeling successfully predicted the temperature dependence of fracture toughness, and explained the central role of micro void nucleation at both micron and submicron-sized particles in an aluminum alloy microstructure. Fracture toughness was governed by particle-matrix interface separation, as well as void-sheet formation from the interplay between temperature-dependent strain/strain rate hardening and resistance to cavitation at 50-100 nm diameter dispersoids within deformation bands. This work was extended to characterize and model the temperature dependence of the fracture toughness of next-generation sub-micron grain sized aluminum alloys produced by rapid-solidification processing. In this alloy class, intravoid strain localization was triggered by strain-rate softening unique to a microstructure consisting of a large volume fraction of 50 nm diameter particles and a lack of dislocation cell structure apart from the submicron grain and sub grain boundaries. The SEM was incapable of effectively imaging the micro void processes at 20-100 nm diameter particles in either alloy class; the models remain speculative pending the application of state-of-the art analysis with an FEG SEM. As a second example, exciting titanium and iron-based alloys have been developed for ultra-high strength and toughness properties. However, such materials are embrittled severely by hydrogen from either processing or environmental exposure. In the former case, research at UVa shows that hydrogen redistributes from moderate-strength trap sites to microstructural locations within the high-hydrostatic stress field at the crack tip, governing the kinetics of embrittlement. The unique result is that such hydrogen diffusion occurs over distances on the

order of 1000 nm and promotes embrittlement at interfaces on the submicron scale. For such complex multi-phase microstructures, very high magnification FEG SEM imaging is required to detail hydrogen damage as a basis for mechanistic modeling. Considering environmental embrittlement, the damage mechanism of either hydrogen enhanced localized plasticity or hydrogen decohesion continues to be controversial. Recent work, employing small-spot STEM, was unable to resolve any segregant that could act synergistically with trapped hydrogen to explain boundary weakening. Very high magnification FEG SEM, using a small spot size and multiple detectors to form secondary-electron images, provided no indication of features on matching intergranular facets that could be traced to localized plasticity. An example is shown in Figure 5, however, such results were limited as the microscope used was outside of the Department of MSE at UVa and access was limited.

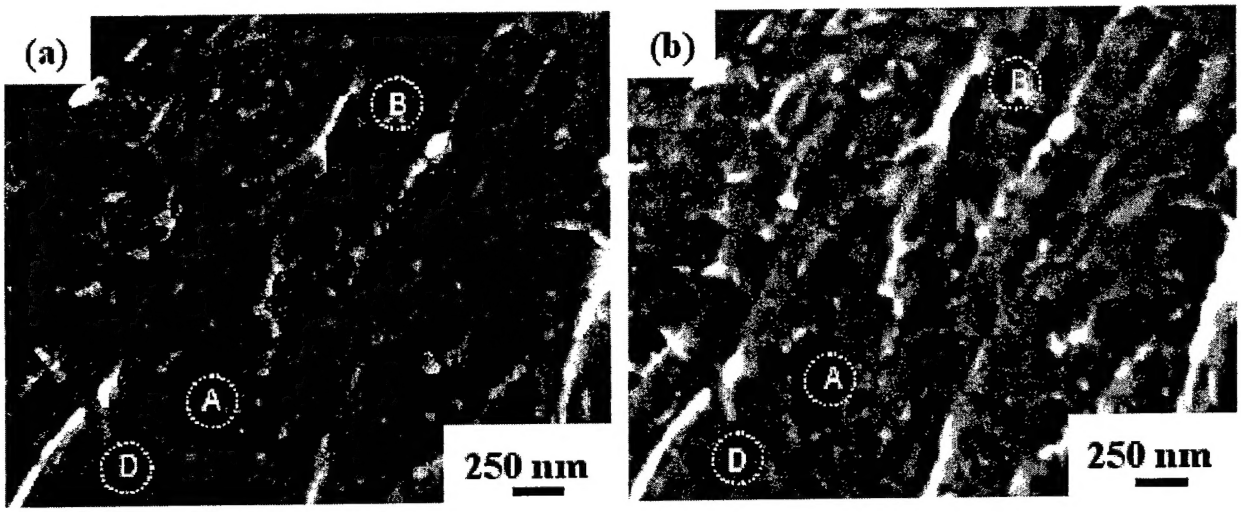

Figure 5. Matching field emission gun SEM fractographs from opposing IG facets for a high strength β-Ti alloy stressed in aqueous NaCl solution. The image in (a) was formed using the secondary electron detector above the specimen, while (b) was formed with both detectors. The relatively featureless facets are interpreted as produced by hydrogen decohesion along a grain boundary surface that contains indications of the underlying α/β-Ti microstructure.

In toto, these observations provide strong support for the ability of trapped hydrogen to lower interface strength leading to embrittlement. Additional work is required, centered on the capabilities provided by the new FEG SEM, to probe and model such processes. For each of the fatigue and fracture problems cited, it is crucial to prepare and examine specimens localized to within 5000 nm of the crack tip and wake surfaces. The new FEG SEM, coupled with the existing FIB and STEM facilities at UVa, will provide an outstanding capability to accomplish such cutting-edge characterizations.

The development of nanostructured materials requires understanding of deformation and fracture mechanisms at yet finer size scales. The approaches taken to attack mechanical behavior deficiencies in modern alloys provide the foundation to understand the mechanical behavior of nanostructures. New deformation and fracture mechanisms will be operative in nanostructures, as demonstrated by the striking difference in the deformation and fracture of conventional *vs* submicron grain size aluminum alloys. Mechanism-based understanding of mechanical damage at the atomic to microscales is the necessary basis for fatigue and fracture mitigation by materials engineering and life prediction at the macroscale. The demonstrated strengths at the University of Virginia, coupled with the new FEG SEM, provide the necessary basis to enable next generation nanostructures that operate at high-performance levels, with predictable reliability and durability.

In addition, John Scully and his graduate students have conducted research on the mechanisms of corrosion of precipitation age hardened alloys as well as amorphous-nanocrystalline materials, as supported by grants from NASA (see above for #), AFOSR [F49620-96-1-0178] (with R.G. Kelly and S.R.Taylor), ONR (with R.P. Gangloff, see above for #), DOE [DEFG02-00ER45825] (with J.L. Hudson, Chem. Eng), NSF[DMR-9357463], and Alcoa Corp (with R.G. Kelly and R.P. Gangloff).

A common aspect of all of these studies has been to identify the metallurgical culprit responsible for the initiation and propagation of extremely localized corrosion and to devise metallurgical, surface treatment, passivation, or inhibition strategies to mitigate these processes. One crucial theme of this work, is to stop or prevent the corrosion process at finer and finer scales since the process is much more difficult to arrest at the micrometer scale. For instance, in AFOSR funded work, our goal is to understand the role of intermetallic compounds (IMC's) on local corrosion. These IMC's are present both as micrometer-scale constituent particles (Al-Fe-Cu, A-Cu-Mg and Al-Cu-Fe-Mn, etc.) and nm-scale precipitate phases (Al-Cu, and Al-Cu-Mg, etc.) in aerospace alloys such as AA 2024-T3. Current understanding is that these phases promote micro- and nano-galvanic couples with the Al-rich matrix, which triggers the onset of localized corrosion (initially nm-scale cathodic trenching or pitting in the alloy matrix next to IMC's, or complex mechanisms of corrosion of the IMC's, themselves). This process occurs under paints and surface films, which is the focus of AFOSR work. Currently, progress has been made in identifying several mechanisms of local corrosion initiation but the susceptibility depends critically upon exact IMC identity. Even small variations in composition within a S-Al_2CuMg and Al-Cu-Fe-Mn-Si phase can alter corrosion kinetics. For instance, Fe and Si may passivate these phases. Therefore, heterogeneous initiation sites for corrosion are also extremely rare and attack does not occur at every IMC. However, our currently used tools such as atomic force microscopy (AFM) and confocal laser scanning microscopy (CLSM) severely limit our metallurgical understandings because they are restricted to only nano- and micro-scale metrological information regarding corrosive damage, albeit in-situ. Scanning auger microscopy provides one alternative that provides surface composition. However, our present information and state of knowledge would be vastly improved if it were coupled with information on local matrix and particle compositions, image analysis information on IMC particle size distributions and crystal orientation information in the f.c.c. Al grains which corrode adjacent to IMC's. Intermetallic composition exerts an overwhelming influence on corrosion properties, yet current SEM-EDS methods using existing U Va. Facilities cannot resolve IMC composition without the risk of electron beam spillover into and X-ray generation from the Al-rich matrix. Therefore, our efforts to understand the details of such corrosion processes are thwarted. Transmission electron microscopy tools do not overcome this problem because TEM foils do not readily contain the variety of intermetallic identities, sizes and compositions present in a large complex engineering alloy, because corrosion initiation is an extremely rare process, and because foil preparation is always an issue in corrosion studies. These research barriers could be overcome by utilization of the new FEG-SEM with its enhanced capabilities in conjunction with our existing methods. Moreover, intergranular corrosion and intergranular stress corrosion cracking (IGSCC) present an even greater scientific challenge with barriers imposed by similar experimental limitations. IGSCC in many precipitation age hardened alloys occurs upon a transition of the nanoscale pitting events described above into focused nm-scale corrosion of nm-sized solute depletion zones along grain boundaries such as Cu-depleted grain and subgrain boundaries. This transition occurs when the pit or cathodic trench intercepts such weakened homophase and heterophase interfaces. The details of this transition process are lost when secondary electron imaging is performed at the 100 nm scale. Advances in the understanding of nm-scale interface corrosion require nm-scale secondary electron resolution combined with grain misorientation information. The lack of this capability impedes progress in understanding IGC and IGSCC.

Perhaps the ultimate corrosion challenge involves the understanding of the mechanism by which small nanocrystals (e.g., < 30 nm) formed in a heat treated amorphous matrix retain the localized corrosion resistance of the amorphous state but lose resistance when grown to larger sizes. The answer likely lies in the complex phases transformation that occur during devitrification including repartitioning of beneficial solute. Specifically, beneficial supersaturated solute is rejected from nanocrystals and can be redistributed into the remaining amorphous matrix. Corrosion pits are presumed to initiate at nanocrystals but these events have never been directly imaged. Nanometer scale pits can be captured by AFM methods but lack of crystallographic and compositional information renders the presumption that the process occurred at a nanocrystal pure speculation. Once again progress is limited by the inability to obtain compositional information, nano-crystal orientation information and secondary electron information with depth resolution from the vicinity of the same pit site. The new FEG-SEM would significantly lower the current barriers to these and other related problems.

Many other unresolved critical issues in corrosion could be advanced by application of this instrument. These include the role of hard IMC's embedded in ductile materials at crack tips in stress corrosion cracking,

understanding of slip steps (e.g., plastic deformation by dislocation motion) on depassivation-repassivation processes in ductile materials and materials containing hard particles, etc. Universally, the main advantage would be simultaneous and coupled composition, structure, and morphological information at finer length scales.

Robert Hull has been working in the field of high-resolution electron microscopy for over 15 years, 10 of which he was a distinguished scientist at Bell Labs, working in the areas of electronic materials, properties and synthesis. Currently, Robert and his students are using high-resolution focused ion beam (FIB) in the development of nanoscale print technologies. This research is sponsored by DARPA under the Molecular Level Printing Program ("Nanoscale Fabrication and Characterization using Focused Electron and Ion Beams", $2,700,000, 09/01/98-08/31/02, N66001-98-1-8917). The core of this program is to use focused ion beam sputtering to create nanoscaled "printheads", the patterns from which are transferred to planar or curved targets via direct contact pattern transfer (microcontact printing, local crystallization, direct imprinting, or seeded anodic oxidation), with inter- and intra-level registration to better than 100 nm. We have succeeded in transferring patterns with 100 nm features, with contact fields up to 1 square mm, and with intra-level registration to tens of nm. In the remaining two years of the program, our goals are to extend this technique to single contact fields of 1 square cm (with total patterned areas of hundreds of square cm through repeated contacting), and both intra- and inter-level registration of 30 nm. These techniques are applicable to a very broad range of materials and surface geometries, with extensive application to high resolution patterning of curved surfaces, rapid prototyping and on-demand lithography. We believe we have made great progress in this program, and will be vigorously pursuing continuing DoD funding for these activities beyond the completion of the current program.

The new FEG SEM and combination lithography system would have extensive applications to this program. When fabricating printheads with feature dimensions on the order of the tens of nanometers, an inspection tool with resolution of nanometers is critical. The FEG SEM is the most efficient and practical tool for providing such resolution. In particular, the ability to image at high resolution with low beam voltage (~ 1 kV, to reduce specimen charging and beam damage) is crucial for imaging the elastomeric molds that are a critical component of the microcontact printing techniques we employ. Such molds are difficult to image in the atomic force microscope, because the scanning tip is liable to deform the elastomer during imaging and distort the features, and specimen preparation for transmission electron microscope is impractical. The fidelity and resolution with which features are transferred from the printhead to the elastomer mold is one of the most critical steps in determining and optimizing the resolution of the microcontact printing technique. We currently observe considerable degradation in feature definition during this step from AFM images, but without high-resolution low voltage SEM imaging we cannot be sure whether this degradation is real or whether it is partially an artifact of the tip-sample interaction in the AFM.

The high-resolution electron lithography capability of this instrumentation will also be of enormous benefit to this project. While the focused ion beam offers many advantages for patterning of the printheads (such as high depth of focus of order 100 um, resistless patterning, and capability for multiple relief), it cannot compete with electron lithography in terms of resolution and throughput. The new field emission electron system will provide us with the capability to extend our printhead technology down to 10 nm. This may open broad new applications such as printing of ultra-dense magnetic nanoparticle arrays for storage applications. Even for applications where 100 nm features are employed, the ability to incorporate much smaller alignment and maker features will be critical to ensuring accurate alignment between successive stampings. In summary, the new field emission electron system would provide substantial additional capabilities to our nanoprinting project, in both microscopy and lithography.

James Fitz-Gerald is currently performing research in areas utilizing advanced laser processing for the last seven years. Before joining the faculty at UVa, he was a research fellow at Naval Research Laboratory working on several DoD projects (Mesoscopic Integrated Conformal Electronics (*MICE)*, a DARPA sponsored program what was administered by ONR, 3/99-9/99, N0001499WX21024, 10/99-9/00 N0001400WR20251, Entitled: "Direct Writing Circuit Elements for a Credit Card GPS by Matrix Assisted Pulsed Laser Evaporation Direct Write") in the following areas: 1) Nanophase materials processing and laser dissociation, 2) Biofunctional polymer thin film synthesis for chemical sensors (warfare and non), 3) Investigation of nanoscale interfaces and composition, 4) Surface modification and nanoscale thin film processing for electronic and corrosion resistant materials. The use of a high-resolution FEG SEM was the only viable method for accurate,

high-resolution morphological characterization of polymer and electronic materials in the above research areas. Chemical sensors for biological warfare detection require nanometer thick films with controlled chemistry involving complex molecules such as SXFA (Poly(oxy{methyl[4-hydroxy-4,4,bis(trifluoromethyl)but-1-en-1-yl}silylene})) to be successfully integrated into useful DoD sensor systems. Using the FEG SEM allowed the investigation of properties such as surface roughness and morphology at high resolution without destroying the sensitive chemical polymer. In addition to chemical sensing, the use of the FEG SEM had significant impact project in the areas of conformal microelectronics and patterning of high-resolution phosphor display systems for heads up display systems (HUDS). In the areas of basic/applied research, the new microscope would enable the PI to continue research on carbon nanotube composites which require the use of an FEG SEM in terms of resolution and charging behavior. Recent research done at a DoD laboratory indicate that it is possible to control the growth of carbon/nanotube composites with laser based processing. Development of this research in areas of nano-chemical sensors, bio-reactive drug delivery, and electronic membranes are currently being pursued at Uva at this time. Figure 6, shown below, illustrates some of the capabilities of the field emission SEM in areas of polymer nanotube composites and membrane applications.

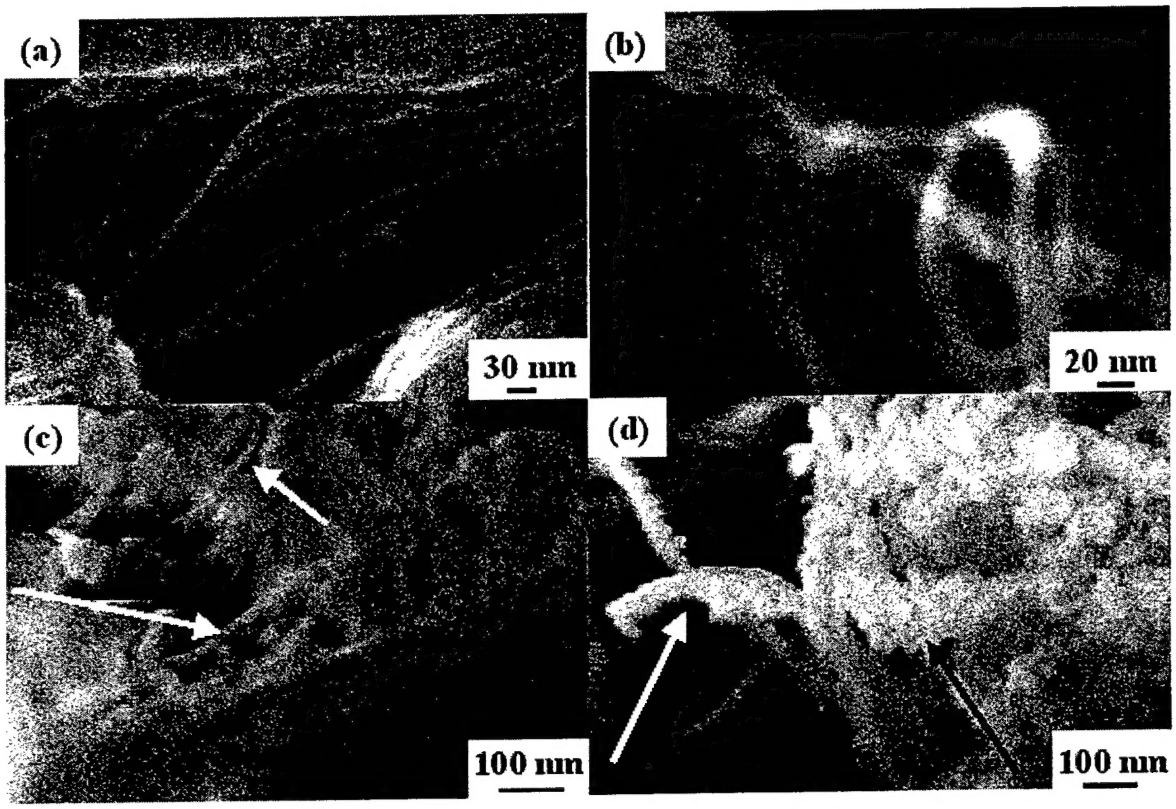

Figure 6. High resolution FEG SEM of carbon nanotube systems, (a) polymer (PEG) – nanotube composite thin film, (b) individual carbon nanotube on the surface, (c) carbon nanotube chemical sensor thin film array, (d) individual carbon nanotubes coated with a chemo-selective polymer. It should be noted that all of the above was accomplished via laser processing.